Recent progress on CO_2 capture using amine-functionalized silica

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Escalating CO_2 emission into the atmosphere is causing great concern about the global climate change. CO_2 capture and storage (CCS) technology is considered the most effective option to alleviate this issue in the short term. Among the various adsorbent materials for CCS, amine-modified silica has been widely studied. In this short review, advances in the past two years on amine-modified silica for CO_2 capture are summarized, and their preparation and performances are discussed.

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Introduction

Anthropogenic CO_2 emissions, mainly generated from the combustion of fossil fuels, have caused great concern about global warming. Since fossil fuels will remain the primary energy resource in the foreseeable future, carbon capture and storage (CCS) seems to be the most promising strategy to alleviate CO_2 emission issue in the short term [1,2].

Liquid phase amine-based CO_2 absorption process has been used in commercial operations throughout the world, where amines capture CO_2 with high selectivity through chemical reactions [3]. However, this gas−liquid absorption process presents several shortcomings, including a high energy requirement for sorbent regeneration and equipment corrosion [4]. Alternatively, porous solid adsorbents such as activated carbons [5], zeolites [6], porous organic polymers [7,8], poly-ionic-liquid (PILs) [9,10], and metal−organic frameworks (MOFs) [11] have been investigated as CO_2 adsorbents [12,13]. However, these materials capture CO_2 through physical adsorption, and consequently have low CO_2 capture capacity and selectivity.

One option to combine the advantages of amines and solid CO_2 adsorbents with high porosity is introducing amines to the pore space of porous materials to form composites [14]. Porous silica has been most frequently utilized as the support material, owing to their outstanding properties such as large surface area/pore volume, adjustable pore size, diversified pore structures, good mechanical stability, and abundant surface hydroxyl groups for organic functionalization [3,15−17]. Several types of amine-silica composites have been prepared via physical impregnation, chemical grafting, or direct single-step methods (Figure 1) for use in CO_2 capture [18−21]. The supported amine could interact with CO_2 via the formation of ammonium carbamates under anhydrous conditions [13]. Loganathan et al. also reported that the primary amine groups interacted more strongly with CO_2 than the secondary or tertiary amine groups, and formed more stable carbomate, because of the steric hindrance of the secondary or tertiary amine [32]. This review summarizes and discusses recent advances in amine-modified porous silica for CO_2 capture. Since several review papers have already been published on CO_2 capture using porous materials [3,13,14], herein we shall focus on research works reported in the past 2 years.

CO_2 capture by amine-impregnated silica

Physical impregnation is the simplest method to load a large amount of amine species into the porous silica support material [3]. However, the stability of the amine-impregnated silica is usually a problem because of amine leaching during long-term operation. To prohibit the amine leaching from the porous silica materials, the impregnated amine should have a high boiling point, and the experimental run should be conducted at relative low temperature [22].

As summarized in Table 1, many different silica materials have been examined as the support for amine impregnation in the last 2 years. Min et al. [23] reported that polyethyleneimine (PEI)/macroporous silica (MacS) showed a higher CO_2 capture capacity of 169 mg/g (40 °C, 0.15 atm) than other mesoporous silica supports such as MCF (mesocellular silica foam), SBA-15, and MCM-41. The large pore volume (1.8 cm^3/g) and pore size (56 nm) of MacS minimize pore obstruction by the amine. After PEI/MacS was placed in steam
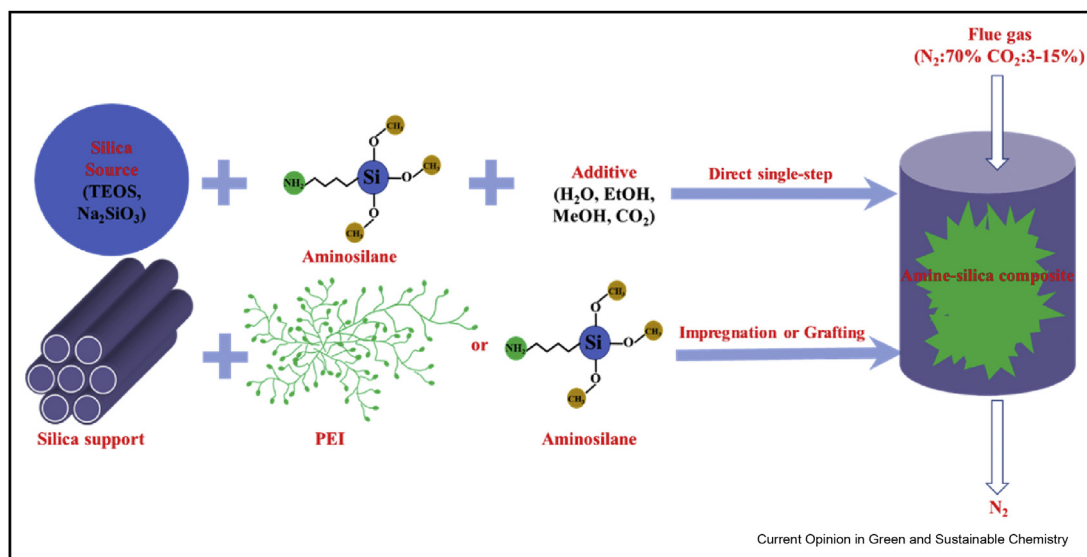
Figure 1

Illustration of the methods to introduce amine species to produce an amine-silica composite for CO_2 capture.

for 14 days at 120 °C, 90% of its CO_2 uptake capacity was maintained. Jiao et al. [24] confirmed that supports with large pores are desirable for improving the CO_2 uptake. Samples of mesoporous silica MSU prepared at 25, 45, and 65 °C were impregnated with tetraethyle-nepentamine (TEPA). The sample synthesized at 65 °C with the largest pores (16.2 nm) showed the highest CO_2 capture performance (189 mg/g) at 25 °C and 1 atm after TEPA-impregnation. Singh et al. [25] found that TEPA-impregnated KCC-1 exhibited high CO_2 uptakes, namely 170 mg/g (dry) and 177 mg/g (humid) at 50 °C and 0.15 atm. This was attributed to the unique fibrous morphology of KCC-1, which resulted in enhanced CO_2 accessibility. Niu et al. [26] pretreated pristine halloy-site nanotubes with heating and acid to produce meso-porous silica nanotubes (MSiNTs) with a high specific surface area (366.4 m²/g) and pore volume (0.55 cm³/g). After combination with PEI, the hybrid PEI/MSiNTs achieved a CO_2 adsorption amount of 121 mg/g at 85 °C and 0.6 atm. Again, a larger pore size/pore volume in the silica support was beneficial for accommodating a high amine loading and facilitating the CO_2 diffusion.

In addition to the silica support, CO_2 capture is also influenced by the types of amine employed. PEI and TEPA have been most frequently studied owing to their high N concentration for high CO_2 uptake and CO_2/N_2 selectivity, and low volatility to alleviate the amine leaching problem [24,27]. As shown in Table 1, PEI molecules with linear and branched structures, and different molecular weights have been investigated. Thakkar et al. [28] proposed a 3D-printing technology to prepare PEI and TEPA-impregnated silica adsor-bents. The prepared TEPA/silica exhibited higher CO_2

uptakes (98 mg/g (dry) and 137 mg/g (humid) at 25 °C and 0.1 atm) than PEI/silica, owing to the higher ni-trogen concentration in TEPA. However, the low mo-lecular weight of TEPA led to relatively lower thermal stability than the PEI/silica during the cyclic adsorption—desorption process. Jung et al. [29] pro-posed forming crosslinks between the impregnated PEI molecules to improve the physical and chemical stability of amine-impregnated silica adsorbents. They used 1,3-butadienediepoxide (BDDE) as a crosslinker because it can react with PEI to convert the primary amine to secondary amine and produce hydroxyl groups. A lower amount of primary amine could avoid urea formation, and the addition of BDDE can increase the PEI vis-cosity, leading to enhanced physical stability of the PEI-impregnated adsorbent.

CO₂ capture by amine-grafted silica
For amine-grafted silica, covalent bonds can be formed between the introduced amine species and the hy-droxyl groups of silica supports via a condensation re-action [13]. Thus, the thermal stability of the amine-grafted silica is superior to that of the amine-impregnated ones. In a typical experiment, silica is dispersed in a solvent (generally anhydrous toluene), then the aminosilanes was added, and finally the resulting mixture solution was heated under reflux. The solid product was recovered by filtering and washed repeatedly to remove the unreacted amino-silanes [3]. Whereas, the amount of amine introduced by grafting is limited by the amount of available hy-droxyl groups, and it is usually lower than that obtained by impregnation. Previously, for impregnation of high

Table 1

**CO₂ capture by amine-functionalized silica.**

Support	Amine type	Temp. (°C)	CO₂ partial pressure (atm)	CO₂ adsorption capacity (mg/g)		Ref.
				Dry CO₂	Humid CO₂	
physical impregnation method						
MCM-41	Polyethyleneimine (Mw~600)	25	1	155	–	[18]
SBA-15	Polyethyleneimine (Mw~5800)	45	1	80	–	[27]
SBA-15	Ethylenediamine-modified poly (vinyl chloride)	25	0.5	22	–	[55]
KIT-6	Polyethyleneimine (Mw~25000)	105	1	106	–	[41]
KIT-6	Pentaethylenehexamine	105	1	197	–	[42]
As-syn KIT-6	Pentaethylenehexamine	75	1	139	–	[20]
H-MSU	Tetraethylenepentamine	25	1	189	–	[24]
HMS	Polyethyleneimine (Mw~800)	90	1	184	–	[21]
Pore-expanded HMS	Tetraethylenepentamine	25	1	150	–	[43]
KCC-1	Tetraethylenepentamine	50	0.15	170	177	[25]
Platelet silica	Polyethyleneimine (Mw~1800)	75	0.1	249	–	[44]
CS-2133 silica	Polyethyleneimine (Mw~2500)	85	0.95	159	–	[45]
Spray-dried silica	Tetraethylenepentamine	30	0.15	88	–	[46]
Mesostructured cellular foam silica	Crosslinker-modified polyethyleneimine (Mw~1200)	40	0.15	107	–	[29]
PD silica	Tetraethylenepentamine	25	0.1	98	137	[28]
Macroporous silica	Polyethyleneimine (Mw~1200)	40	0.15	–	169	[23]
Trimodal nanoporous silica	Polyethyene polyamine	75	1	172	–	[47]
Hierarchically porous silica	Polyethyleneimine (Mw~600)	75	1	107	–	[48]
Hydrophobic silica	Polyethyleneimine (Mw~2500)	25	0.1	–	130	[49]
Mesoporous silica nanotubes	Polyethyleneimine (Mw~800)	85	0.6	121	–	[26]
Precipitated silica	3-aminopropyltriethoxysilane	70	1	80	–	[40]
Mesoporous multilamellar silica vesicle (with template)	Polyethylenimine (Mw~600)	75	1	179	219	[50]
Rice husk derived silica	Polyethyleneimine (Mw~600)	75	0.15	159	–	[51]
Grade Q-10 silica	Polyethylenimine (Mw~800)	75	0.1	102	–	[52]
Core-shell 5A@mesoporous silica	Polyethylenimine (Mw~600)	25	0.15	72	222	[53]
Silica gel grafted with N¹-(3-trimethoxysilylpropyl) diethylenetriamine	Tetraethylenepentamine	75	1	116	–	[62]
Mesoporous silica	Polyethyleneimine (Mw~1800)	70	0.15	97	100	[54]
chemical grafting method						
Pore-expanded MCM-41	N-(3-trimethoxysilylpropyl) diethylenetriamine	75	0.2	92	–	[31]
Pore-expanded MCM-41	N-(3-trimethoxysilylpropyl) diethylenetriamine	75	0.2	92	–	[32]
MCM-48	2-[2-(3-trimethoxysilylpropylamino) ethylamino] ethylamine	25	0.15	70	–	[56]
SBA-15	3-[2-(2-Aminoethylamino) ethylamino] propyl trimethoxysilane	25	0.05	83	–	[19]
SBA-15	N-propyl ethylenediamine	0	1	78	–	[57]
SBA-15	(3-Aminopropyl) triethoxysilane	25	10	88	–	[58]
SBA-15	3-[2-(2-Aminoethylamino)ethylamino]propyl trimethoxysilane	25	0.05	83	–	[34]
HMS	Aminopropyltrimethoxysilane	20	0.9	53	–	[37]
HMS@Mg-Al LDH	N1-(3-Trimethoxysilylpropyl)diethylenetriamine	25	1	56	–	[59]
P10 silica	3-[2-(2-Aminoethylamino)ethylamino] propyltrimethoxysilane	50	0.05	86	–	[33]
Ordered mesoporous silica	Lysine functionalized 1-methyl-3-ethyl-imidazolium based ionic liquid	25	1	27	–	[35]
Amorphous silica	[3-(methylamino)propyl]trimethoxysilane	30	0.17	60	–	[36]
one-step method						
Precipitated silica	(3-Aminopropyl) triethoxysilane	50	0.14	45	–	[38]
Silsesquioxane aerogel	(3-Aminopropyl) triethoxysilane	50	0.01	134	169	[39]
Silica spheres	Trimethoxy [3-(methylamino)propyl]silane	50	0.1	44	–	[60]
Mesoporous Silica	Polyethenimine (branched, Mw~600)	75	0.15	124	–	[61]

amine amounts, triamine with high N concentration was chosen as the amine source, whereas the disadvantage of employing triamine is the occurrence of pore blockage and plugging because of its large molecular size [30]. Compared to the conventional amine grafting, the amine amount on the silica surface could be increased via a wet grafting method [19,31–34]. In the dry amine grafting process, 3 mol hydroxyl groups on the silica support is expected to react with 1 mol aminosilanes (Figure 2(a)) [31]. However, in most cases, the reaction of hydroxyl groups is not complete because of steric hindrance (Figure 2(b)). In the wet grafting method, H_2O can increase the hydroxyl group density on the silica surface and thereby the amount of grafted amine species (Figure 2(c)). The reaction may

also take place between the introduced aminosilanes through siloxane linkages during the wet grafting process (Figure 2(d)), which is also beneficial for increasing the amine loading.

Similar to the cases of amine impregnation, the CO_2 uptake of an amine-grafted porous silica is also influenced by the pore size of the support. Fayaz et al. [33] reported that a larger pore size could increase the CO_2 uptake, and a CO_2 uptake of 86 mg/g at 50 °C and 0.05 atm was achieved with P10 silica with the pore size of 21 nm after functionalization with 3-[2-(2-aminoethylamino)ethylamino] propyltrimethoxysilane. Lashaki et al. [34] also confirmed that the increase of pore size of the silica support was beneficial for

Figure 2
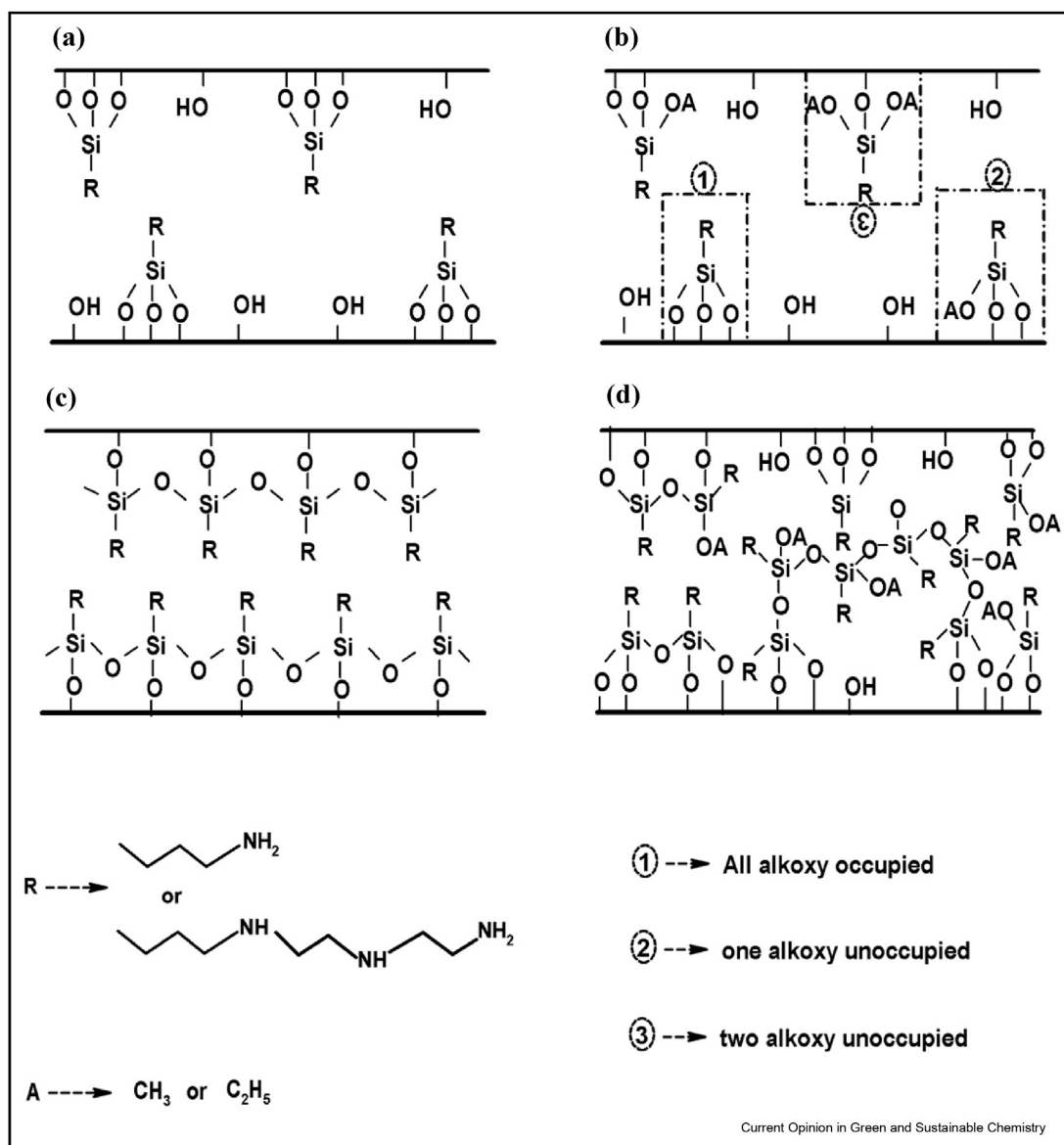
Hypothetical representation of pore wall surface under (a) and (b): dry grafting and (c) and (d): wet grafting conditions [31].

increasing the CO_2 adsorption capacity. 3-[2-(2-Amino-ethylamino) ethylamino] propyltrimethoxysilane was grafted on six kinds of SBA-15 with variable pore sizes and pore volumes. The highest CO_2 uptake (up to 83 mg/g) was achieved by the amine-grafted SBA-15 with big pore size of 10.9 nm at 25 °C and 0.05 bar, whereas further increase of the pore size of SBA-15 (14.5 nm) led to a lower CO_2 uptake due to the increase of pore blockage in the distorted pore structure.

Some new methods were also applied for preparing amine-grafting adsorbents. Hiremath et al. [35] grafted ordered mesoporous silica (OMS) with four different ionic liquids (ILs) derived from amino acids: glycine (Gly), lysine (Lys), proline (Pro), and alanine (Ala). The OMS-IL (Lys) showed a relatively high CO_2 uptake of 27 mg/g at 25 °C and 1 atm, and DFT calculations indicated the highest CO_2 adsorption energy (-0.98 eV) for Lys among the four adsorbents. Moreover, the thermodynamic parameters of OMS-IL (Lys) exhibits the negative enthalpy value (-370.9 kJ/mol) and the positive entropy value (4.57 J/mol K), which indicates that the CO_2 adsorption is an exothermic reaction with high affinity of the adsorbents to CO_2. Nevertheless, the CO_2 uptake amount was still lower than the observed with other amine-grafted silica adsorbents. Park et al. [36] prepared amine-grafted porous silica via an in-situ polymerization method and achieved an amine content in

the adsorbent (2.93 mmol/g) about six times larger than that prepared by conventional grafting method. Knowles et al. [37] grafted aminopropyltrimethoxysilane (APTS) on an HMS silica support via a sonication method, and the method improved both amine dispersion in the solvent and gas diffusion throughout the support pores, leading to increased CO_2 uptakes.

CO₂ capture by amine-modified silica prepared by direct single-step method

The CO_2 capture performances of amine-modified silica prepared via direct single-step methods are also included in Table 1. Quang et al. [38] prepared an APTES-functionalized precipitated silica via a single-step method. As shown in Figure 3, after initial mixing with water, APTES was hydrolyzed (eq. (1)) and the hydrolysis product underwent self-condensation reaction (eq. (2)). The solution was then bubbled with CO_2 to form carbamates (eq. (3)). A silica source was added to form the APTES hydrolysis product and $Si(OH)_4$ (eq. (4)), which then condensed to produce an amine-functionalized silica framework (eq. (5)). The prepared amine-modified silica exhibited a CO_2 uptake of 45 mg/g at 50 °C and 0.14 atm. Kong et al. [39] prepared an APTES-modified silsesquioxane aerogel via the sol–gel method followed by supercritical drying to achieve a CO_2 uptake of 134 mg/g (dry) and 169 mg/g (humid) at 50 °C and 0.01 atm.

Figure 3
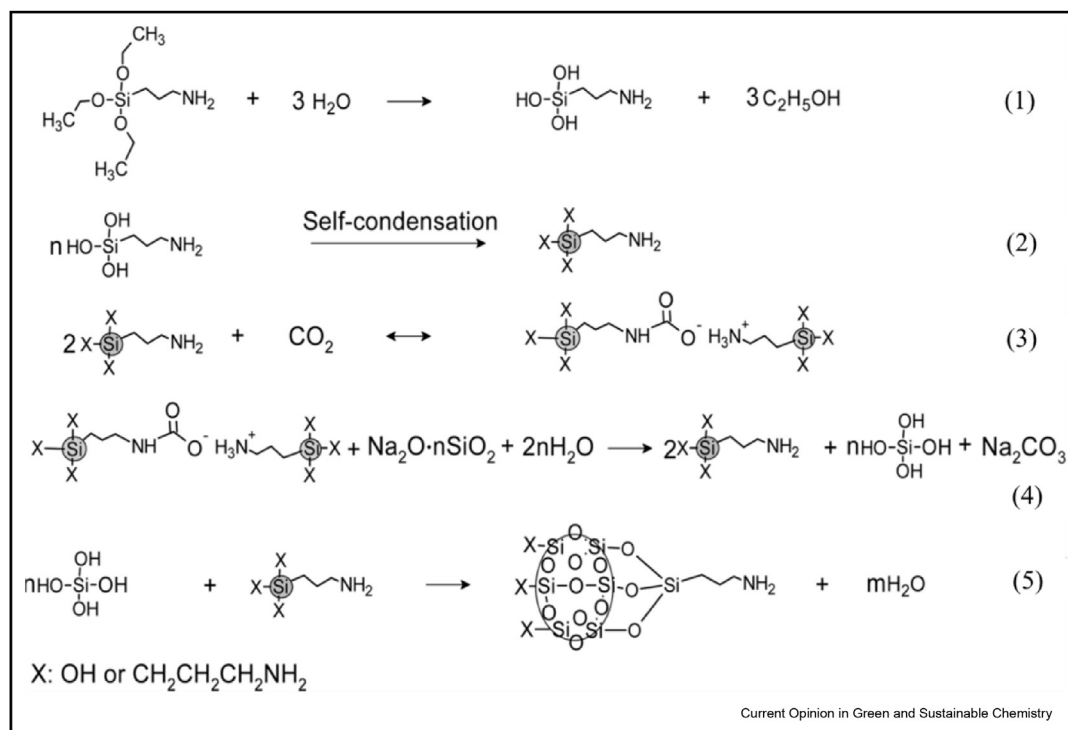
Direct synthesis route of an amine-functionalized silica adsorbent [38].

Conclusions

Amine-modified silicas are promising CO_2 adsorbents exhibiting high CO_2 uptakes and high CO_2/N_2 selectivity, as well as good performance in the presence of moisture. In the past two years, a series of amine-modified silica have been prepared by physical impregnation, chemical grafting, or direct one-step methods for use in CO_2 capture. The amine-impregnated silica is easy to prepare with high amine loadings, while the amine leaching and degradation problem during CO_2 adsorption—desorption runs need to be resolved. Introducing amine with high boiling point or decreasing the operation temperature are potential solutions. Amine-grafted silica, on the other hand, has good stability during adsorption—desorption cycles, but the amine loading is relatively low, which can be improved by employing a wet grafting method. In both the impregnated and grafted systems, the textural properties of silica support and amine type significantly affect the CO_2 capture. Meanwhile, there have been only a few reports on the direct one-step synthesis of amine-modified silicas, which should be considered further in the future.

Conflict of interest statement

Nothing declared.

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